Catalytic molecular motors

Vicario, J; Eelkema, R; Browne, WR; Meetsma, A; Crois, René M. La; Feringa, Bernard

Published in:
Chemical Communications

DOI:
10.1039/b505092h

Publication date:
2005

data_c22.50h2
_database_code_depnum_ccdc_archive 'CCDC 268595'

#==

5. CHEMICAL DATA

_chemical_name_systematic
;
?
;
_chemical_name_common ?
_chemical_melting_point ?
_chemical_formula_moiety 'C45 H41 Mn2 N6 O4, Cl O4'
_chemical_formula_structural ?
Ex: 'C12 H16 N2 O6, H2 O' and '(Cd 2+)3, (C6 N6 Cr 3-)2, 2(H2 O)'
_chemical_formula_sum 'C45 H41 Cl Mn2 N6 O8'
_chemical_formula_weight 939.18
_chemical_compound_source 'by syntheses'

loop_
_atom_type_symbol
_atom_type_description
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_source
O O .0106 .0060 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
N N .0061 .0033 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Mn Mn .3368 .7283 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
Cl Cl .1484 .1585 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
H H .0000 .0000 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

```
C C .0033 .0016 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

#===============================================================================

# 6. CRYSTAL DATA

_symmetry_cell_setting Monoclinic
_symmetry_space_group_name_Hall 'C 2y'
_symmetry_space_group_name_H-M 'C 2'

loop_
_symmetry_equiv_pos_as_xyz
x,y,z
-x,y,-z
1/2+x,1/2+y,z
1/2-x,1/2+y,-z

_cell_length_a 15.226(2)
_cell_length_b 12.924(2)
_cell_length_c 10.944(2)
_cell_angle_alpha 90
_cell_angle_beta 90.157(9)
_cell_angle_gamma 90
_cell_volume 2153.6(6)
_cell_formula_units_Z 2
_cell_measurement_temperature 130
_cell_measurement_reflns_used 22
_cell_measurement_theta_min 16.65
_cell_measurement_theta_max 20.21
_cell_special_details
;
Unit cell parameters (Duisenberg, 1992) and orientation matrix were determined
from a least-squares treatment of SET4 (de Boer & Duisenberg, 1984) setting.
Reduced cell calculations did not indicate any higher metric lattice symmetry
and examination of the final atomic coordinates of the structure did not yield
extra symmetry elements (Spek, 1988; Le Page 1987, 1988)
;

_exptl_crystal_description 'cubic-shaped block'
_exptl_crystal_colour 'light green transparent'
_exptl_crystal_size_max 0.25
_exptl_crystal_size_mid 0.25
_exptl_crystal_size_min 0.25
_exptl_crystal_size_rad ?
_exptl_crystal_density_meas ?
_exptl_crystal_density_diffrn 1.448
_exptl_crystal_density_method ?
_exptl_crystal_F_000 968
_exptl_absorpt_coefficient_mu .71
_exptl_crystal_density_meas_temp ?
_exptl_absorpt_correction_type none
_exptl_absorpt_process_details ?
_exptl_absorpt_correction_T_min ?
_exptl_absorpt_correction_T_max ?

#===============================================================================

# 7. EXPERIMENTAL DATA

_exptl_special_details
;
?
;
_diffrn_ambient_temperature 130
_diffrn_radiation_wavelength .71073
_diffrn_radiation_type 'Mo K\a'
```

```
_diffrn_radiation_source 'fine focus sealed Philips Mo tube '
_diffrn_radiation_monochromator 'perpendicular mounted graphite'
_diffrn_radiation_detector
;
scintillation NaI crystal with photomultiplier
;
_diffrn_measurement_device_type
;
Enraf Nonius CAD-4F diffractometer
;

_diffrn_measurement_method \w/2\q

_diffrn_special_details
;
Crystal into the cold nitrogen stream of the low-temperature unit
(Bolhuis, 1971), on an Enraf-Nonius CAD-4F diffractometer.
Graphite-monochromated Mo K\a radiation,
\w/2\q scan, \D\w = (0.80 + 0.34 tg \q)\%.
;
_diffrn_detector_area_resol_mean ?

_diffrn_standards_number 3
_diffrn_standards_interval_count ?
_diffrn_standards_interval_time 180
_diffrn_standards_decay_% 3.1

loop_
_diffrn_standard_refln_index_h
_diffrn_standard_refln_index_k
_diffrn_standard_refln_index_l
1 -1 -3
2 2 0
4 0 1

# number of measured reflections (redundant set)
_diffrn_reflns_number 5221
_diffrn_reflns_av_R_equivalents 0.0111
_diffrn_reflns_av_sigmaI/netI 0.0159
_diffrn_reflns_limit_h_min -19
_diffrn_reflns_limit_h_max 19
_diffrn_reflns_limit_k_min -16
_diffrn_reflns_limit_k_max 16
_diffrn_reflns_limit_l_min -13
_diffrn_reflns_limit_l_max 0
_diffrn_reflns_theta_min 1.86
_diffrn_reflns_theta_max 26.99
_diffrn_reflns_reduction_process
;
Intensity data were corrected for Lorentz and polarization effects, scale
variation, but not for absorption and reduced to F~o~^2^
;

# number of unique reflections
_reflns_number_total 4691
_reflns_number_gt 4481
_reflns_threshold_expression >2sigma(I)


_computing_data_collection 'CAD4-UNIX Software Version 5.1, 1994'
_computing_cell_refinement 'SET4 (de Boer & Duisenberg, 1984)'
_computing_data_reduction 'HELENA (Spek, 1993)'
_computing_structure_solution
;
DIRDIF-97 (Beurskens et al., 1997)
;
```

```
_computing_structure_refinement 'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics
;
PLUTO (Meetsma, 1997)
ORTEP (Burnett et al., 1996)
PLATON (Spek, 1994, 1996)
;
_computing_publication_material 'PLATON (Spek, 1990)'

#===============================================================================

# 8. REFINEMENT DATA

_refine_special_details
;
Refinement of F^2^ against ALL reflections. The weighted R-factor wR and
goodness of fit S are based on F^2^, conventional R-factors R are based
on F, with F set to zero for negative F^2^. The threshold expression of
F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement. R-factors based
on F^2^ are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
;

_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type full
_refine_ls_weighting_scheme
'calc w=1/[\s^2^(Fo^2^)+(0.0422P)^2^+2.3179P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary heavy
_atom_sites_solution_secondary difmap
_atom_sites_solution_hydrogens difmap
_refine_ls_hydrogen_treatment mixed
_refine_ls_extinction_method none
_refine_ls_extinction_coef ?
_refine_ls_abs_structure_details 'Flack H D (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack 0.428(15)
_refine_ls_number_reflns 4691
_refine_ls_number_parameters 375
_refine_ls_number_restraints 1
_refine_ls_R_factor_all 0.0329
_refine_ls_R_factor_gt 0.0303
_refine_ls_wR_factor_ref 0.0778
_refine_ls_wR_factor_gt 0.0760
_refine_ls_goodness_of_fit_ref 1.027
_refine_ls_restrained_S_all 1.026
_refine_ls_shift/su_max 0.001
_refine_ls_shift/su_mean 0.000

_refine_diff_density_max .551
_refine_diff_density_min -.520
_refine_diff_density_rms .051

#===============================================================================

# 9. ATOMIC COORDINATES AND THERMAL PARAMETERS

loop_
_atom_site_label
_atom_site_thermal_displace_type
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_U_iso_or_equiv
Mn Uani .07875(2) .00111(2) .41011(3) 1.000 .0241(1)
O1 Uani .05356(11) -.04469(14) .59691(14) 1.000 .0301(5)
```

```
O2 Uani .04933(13) .16203(14) .42542(18) 1.000 .0368(6)
N1 Uani .22262(11) -.00802(18) .46914(15) 1.000 .0248(5)
N2 Uani .16141(14) .07360(16) .24962(17) 1.000 .0289(6)
N3 Uani .13522(15) -.14282(15) .31559(18) 1.000 .0305(6)
C1 Uani .10637(15) -.04535(18) .6956(2) 1.000 .0259(6)
C2 Uani .07153(17) -.02851(19) .8126(2) 1.000 .0311(7)
C3 Uani .12479(18) -.03113(19) .9157(2) 1.000 .0327(7)
C4 Uani .21384(18) -.04843(19) .9051(2) 1.000 .0313(7)
C5 Uani .24910(16) -.06486(18) .7896(2) 1.000 .0279(7)
C6 Uani .19682(15) -.06438(17) .68488(19) 1.000 .0248(6)
C7 Uani .23652(17) -.09017(19) .5627(2) 1.000 .0272(7)
C8 Uani .25689(18) .0923(2) .5127(2) 1.000 .0314(8)
C9 Uani .26415(17) -.03766(18) .3515(2) 1.000 .0283(7)
C10 Uani .24612(17) .04740(19) .2596(2) 1.000 .0290(7)
C11 Uani .3115(2) .0945(2) .1919(2) 1.000 .0353(8)
C12 Uani .2879(2) .1746(2) .1141(2) 1.000 .0418(9)
C13 Uani .2009(2) .2028(2) .1043(2) 1.000 .0385(8)
C14 Uani .1392(2) .15075(19) .1740(2) 1.000 .0346(8)
C15 Uani .22261(18) -.13645(18) .3031(2) 1.000 .0299(7)
C16 Uani .2720(2) -.2106(2) .2432(2) 1.000 .0394(9)
C17 Uani .2290(3) -.2947(2) .1941(2) 1.000 .0479(11)
C18 Uani .1395(3) -.3023(2) .2053(2) 1.000 .0485(12)
C19 Uani .0944(2) -.2250(2) .2670(2) 1.000 .0402(9)
C20 Uani .00000 .2060(3) .50000 1.000 .0305(10)
C21 Uani .00000 .3227(3) .50000 1.000 .0416(13)
C22 Uani .01555(18) .3763(3) .3925(4) 1.000 .0535(12)
C23 Uani .0147(2) .4842(3) .3941(6) 1.000 .0804(19)
C24 Uani .00000 .5373(4) .50000 1.000 .100(3)
Cl Uani .00000 .42958(9) .00000 1.000 .0390(3)
O3 Uani .0701(4) .3798(6) -.0492(7) 1.000 .195(3)
O42 Uani .0385(7) .4529(11) .1085(11) .605(17) .161(5)
O41 Uani .0200(11) .5295(11) .0434(11) .395(17) .115(6)
H2 Uiso .009(2) -.010(3) .821(3) 1.000 .056(9)
H3 Uiso .093(2) -.018(3) 1.000(3) 1.000 .069(11)
H4 Uiso .257(2) -.046(3) .984(3) 1.000 .046(9)
H5 Uiso .311(2) -.080(3) .782(3) 1.000 .037(8)
H7 Uiso .2993(18) -.101(2) .577(2) 1.000 .020(6)
H7' Uiso .2100(18) -.152(2) .527(3) 1.000 .024(6)
H8 Uiso .2441(19) .150(3) .458(3) 1.000 .033(7)
H8' Uiso .324(2) .089(3) .524(3) 1.000 .044(9)
H8" Uiso .228(2) .102(3) .600(3) 1.000 .045(9)
H9 Uiso .3258(19) -.047(2) .364(2) 1.000 .029(7)
H11 Uiso .374(2) .071(3) .201(3) 1.000 .047(9)
H12 Uiso .331(3) .204(3) .074(4) 1.000 .062(11)
H13 Uiso .184(2) .259(3) .055(3) 1.000 .046(9)
H14 Uiso .071(2) .172(3) .172(3) 1.000 .042(8)
H16 Uiso .330(3) -.201(4) .227(4) 1.000 .077(14)
H17 Uiso .258(3) -.348(4) .152(4) 1.000 .073(12)
H18 Uiso .108(3) -.351(4) .178(4) 1.000 .066(12)
H19 Uiso .031(2) -.227(2) .276(3) 1.000 .033(7)
H22 Uiso .029(2) .342(3) .334(3) 1.000 .049(11)
H23 Uiso .023(3) .513(4) .329(4) 1.000 .065(12)
H24 Uiso .00000 .616(7) .50000 1.000 .11(3)

loop_
_atom_site_aniso_label
_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
Mn .0288(2) .0259(2) .0177(1) -.0006(1) -.0019(1) .0006(2)
O1 .0312(8) .0421(9) .0170(7) .0003(6) -.0034(6) .0072(7)
O2 .0407(10) .0274(9) .0424(10) .0026(8) .0084(8) .0040(7)
N1 .0321(8) .0225(9) .0197(7) -.0030(8) .0001(6) .0014(9)
```

```
N2 .0424(12) .0230(9) .0212(9) -.0008(7) .0022(8) .0002(8)
N3 .0459(12) .0250(10) .0206(9) -.0031(7) -.0011(8) -.0025(9)
C1 .0339(12) .0251(10) .0187(10) .0008(8) -.0040(9) .0043(9)
C2 .0355(12) .0388(14) .0189(10) -.0001(9) -.0003(9) .0055(9)
C3 .0478(14) .0324(12) .0179(10) -.0023(8) -.0023(9) .0023(10)
C4 .0459(14) .0264(12) .0216(11) .0022(9) -.0108(10) -.0019(10)
C5 .0343(12) .0254(11) .0241(11) .0051(9) -.0040(9) .0012(9)
C6 .0324(11) .0221(10) .0198(10) .0006(8) -.0041(8) .0035(9)
C7 .0317(12) .0275(12) .0225(12) .0006(9) -.0013(9) .0070(9)
C8 .0373(14) .0289(13) .0281(13) -.0059(10) -.0029(10) -.0019(10)
C9 .0332(12) .0253(10) .0263(12) -.0026(9) .0024(9) .0011(9)
C10 .0434(14) .0251(11) .0185(10) -.0051(9) .0002(9) -.0024(10)
C11 .0450(15) .0388(14) .0222(11) -.0040(10) .0068(10) -.0068(11)
C12 .0619(18) .0391(15) .0243(12) .0007(11) .0082(12) -.0155(13)
C13 .0663(19) .0265(12) .0228(11) .0016(10) -.0011(12) -.0052(12)
C14 .0583(17) .0259(11) .0197(11) -.0025(9) -.0022(10) .0012(11)
C15 .0478(14) .0248(11) .0170(10) .0008(8) .0033(9) .0043(10)
C16 .0666(19) .0269(13) .0248(12) .0046(9) .0117(12) .0100(12)
C17 .097(3) .0239(12) .0229(12) .0010(10) .0154(14) .0052(14)
C18 .100(3) .0222(12) .0232(13) -.0031(10) -.0009(14) -.0128(14)
C19 .067(2) .0298(13) .0237(12) .0012(10) -.0031(12) -.0129(13)
C20 .0212(15) .0259(16) .0444(19) .0000 -.0022(13) .0000
C21 .0155(15) .0252(17) .084(3) .0000 .0082(17) .0000
C22 .0198(12) .0357(15) .105(3) .0176(18) .0148(15) .0023(11)
C23 .0304(14) .036(2) .175(5) .038(2) .037(2) .0064(13)
C24 .030(2) .025(2) .246(10) .0000 .053(4) .0000
Cl .0301(4) .0532(6) .0337(5) .0000 .0001(3) .0000
O3 .138(4) .220(7) .226(6) .006(5) .028(4) .131(5)
O42 .166(7) .158(11) .158(8) -.039(8) -.127(7) .012(7)
O41 .157(14) .104(9) .084(8) -.049(6) .024(7) -.098(10)

#===============================================================================

# 10. MOLECULAR GEOMETRY

_geom_special_details
;
Bond distances, angles etc. have been calculated using the
rounded fractional coordinates. All esds are estimated
from the variances of the (full) variance-covariance matrix.
The cell esds are taken into account in the estimation of
distances, angles and torsion angles
;

loop_
_geom_bond_atom_site_label_1
_geom_bond_atom_site_label_2
_geom_bond_distance
_geom_bond_site_symmetry_1
_geom_bond_site_symmetry_2
_geom_bond_publ_flag
Mn O1 2.1636(16) . . yes
Mn O2 2.1341(19) . . yes
Mn N1 2.2852(17) . . yes
Mn N2 2.358(2) . . yes
Mn N3 2.297(2) . . yes
Mn O1 2.1009(18) . 2_556 yes
Cl O41 1.409(14) . . yes
Cl O3 1.359(7) . . yes
Cl O42 1.357(12) . . yes
O1 C1 1.345(3) . . yes
O2 C20 1.248(3) . . yes
O41 O41 1.127(19) . 2_555 yes
N1 C8 1.476(3) . . yes
N1 C9 1.486(3) . . yes
N1 C7 1.490(3) . . yes
```

```
N2 C10 1.338(3) . . yes
N2 C14 1.339(3) . . yes
N3 C19 1.340(3) . . yes
N3 C15 1.341(4) . . yes
C1 C6 1.404(3) . . no
C1 C2 1.404(3) . . no
C2 C3 1.388(3) . . no
C3 C4 1.379(4) . . no
C4 C5 1.391(3) . . no
C5 C6 1.394(3) . . no
C6 C7 1.506(3) . . no
C9 C15 1.520(3) . . no
C9 C10 1.515(3) . . no
C10 C11 1.384(4) . . no
C11 C12 1.387(4) . . no
C12 C13 1.378(4) . . no
C13 C14 1.386(4) . . no
C15 C16 1.384(4) . . no
C16 C17 1.377(4) . . no
C17 C18 1.372(6) . . no
C18 C19 1.389(4) . . no
C20 C21 1.508(5) . . no
C21 C22 1.386(5) . . no
C22 C23 1.395(5) . . no
C23 C24 1.366(6) . . no
C2 H2 .99(3) . . no
C3 H3 1.06(3) . . no
C4 H4 1.08(3) . . no
C5 H5 .97(3) . . no
C7 H7 .98(3) . . no
C7 H7' .98(3) . . no
C8 H8" 1.06(3) . . no
C8 H8 .98(4) . . no
C8 H8' 1.03(3) . . no
C9 H9 .96(3) . . no
C11 H11 1.00(3) . . no
C12 H12 .88(4) . . no
C13 H13 .94(4) . . no
C14 H14 1.07(3) . . no
C16 H16 .91(5) . . no
C17 H17 .94(5) . . no
C18 H18 .85(5) . . no
C19 H19 .97(3) . . no
C22 H22 .81(3) . . no
C23 H23 .81(5) . . no
C24 H24 1.02(9) . . no

loop_
_geom_angle_atom_site_label_1
_geom_angle_atom_site_label_2
_geom_angle_atom_site_label_3
_geom_angle
_geom_angle_site_symmetry_1
_geom_angle_site_symmetry_2
_geom_angle_site_symmetry_3
_geom_angle_publ_flag
O1 Mn O2 98.90(7) . . . yes
O1 Mn N1 83.74(6) . . . yes
O1 Mn N2 155.37(7) . . . yes
O1 Mn N3 105.76(7) . . . yes
O1 Mn O1 77.59(6) . . 2_556 yes
O2 Mn N1 103.26(8) . . . yes
O2 Mn N2 77.52(7) . . . yes
O2 Mn N3 154.64(7) . . . yes
O1 Mn O2 94.36(7) 2_556 . . yes
N1 Mn N2 73.61(7) . . . yes
```

```
N1 Mn N3 74.08(8) . . . yes
O1 Mn N1 156.08(7) 2_556 . . yes
N2 Mn N3 77.59(7) . . . yes
O1 Mn N2 126.76(7) 2_556 . . yes
O1 Mn N3 96.63(7) 2_556 . . yes
O3 Cl O41 118.0(7) . . 2_555 yes
O3 Cl O42 95.5(6) 2_555 . . yes
O42 Cl O42 154.3(9) . . 2_555 yes
O3 Cl O42 96.5(6) . . . yes
O3 Cl O41 113.5(7) . . . yes
O3 Cl O3 123.5(5) . . 2_555 yes
O3 Cl O42 95.5(6) . . 2_555 yes
O3 Cl O41 118.0(7) 2_555 . . yes
O41 Cl O41 47.1(8) . . 2_555 yes
O3 Cl O42 96.5(6) 2_555 . 2_555 yes
O3 Cl O41 113.5(7) 2_555 . 2_555 yes
Mn O1 Mn 93.49(7) . . 2_556 yes
Mn O1 C1 127.04(14) 2_556 . . yes
Mn O1 C1 130.80(14) . . . yes
Mn O2 C20 128.5(2) . . . yes
Cl O41 O41 66.4(10) . . 2_555 yes
Mn N1 C9 100.27(13) . . . yes
C7 N1 C9 110.59(19) . . . yes
C8 N1 C9 110.81(18) . . . yes
C7 N1 C8 110.77(17) . . . yes
Mn N1 C7 111.43(14) . . . yes
Mn N1 C8 112.56(15) . . . yes
C10 N2 C14 118.7(2) . . . yes
Mn N2 C10 110.80(15) . . . yes
Mn N2 C14 128.47(18) . . . yes
C15 N3 C19 117.9(2) . . . yes
Mn N3 C19 130.31(19) . . . yes
Mn N3 C15 111.66(15) . . . yes
O1 C1 C6 121.23(19) . . . yes
C2 C1 C6 118.4(2) . . . no
O1 C1 C2 120.3(2) . . . yes
C1 C2 C3 121.1(2) . . . no
C2 C3 C4 120.5(2) . . . no
C3 C4 C5 118.9(2) . . . no
C4 C5 C6 121.7(2) . . . no
C1 C6 C7 120.6(2) . . . no
C5 C6 C7 120.0(2) . . . no
C1 C6 C5 119.4(2) . . . no
N1 C7 C6 113.32(19) . . . yes
N1 C9 C10 108.14(19) . . . yes
N1 C9 C15 109.9(2) . . . yes
C10 C9 C15 107.69(19) . . . no
C9 C10 C11 123.1(2) . . . no
N2 C10 C11 122.7(2) . . . yes
N2 C10 C9 114.3(2) . . . yes
C10 C11 C12 118.1(3) . . . no
C11 C12 C13 119.5(3) . . . no
C12 C13 C14 118.8(2) . . . no
N2 C14 C13 122.1(3) . . . yes
N3 C15 C9 115.4(2) . . . yes
N3 C15 C16 123.1(2) . . . yes
C9 C15 C16 121.4(2) . . . no
C15 C16 C17 118.2(3) . . . no
C16 C17 C18 119.5(3) . . . no
C17 C18 C19 119.0(3) . . . no
N3 C19 C18 122.3(3) . . . yes
O2 C20 O2 125.8(3) . . 2_556 yes
O2 C20 C21 117.10(18) 2_556 . . yes
O2 C20 C21 117.10(18) . . . yes
C22 C21 C22 120.0(4) . . 2_556 no
C20 C21 C22 120.0(2) . . 2_556 no
```

```
C20 C21 C22 120.0(2) . . . no
C21 C22 C23 119.2(4) . . . no
C22 C23 C24 121.0(5) . . . no
C23 C24 C23 119.7(5) . . 2_556 no
C1 C2 H2 119.3(19) . . . no
C3 C2 H2 119.5(19) . . . no
C4 C3 H3 123.5(17) . . . no
C2 C3 H3 116.0(17) . . . no
C3 C4 H4 121.5(17) . . . no
C5 C4 H4 119.6(17) . . . no
C4 C5 H5 119(2) . . . no
C6 C5 H5 119(2) . . . no
C6 C7 H7 106.5(13) . . . no
C6 C7 H7' 111.7(18) . . . no
H7 C7 H7' 110(2) . . . no
N1 C7 H7 110.4(14) . . . no
N1 C7 H7' 104.5(18) . . . no
H8 C8 H8' 108(3) . . . no
H8 C8 H8" 112(3) . . . no
H8' C8 H8" 108(2) . . . no
N1 C8 H8" 104(2) . . . no
N1 C8 H8 114(2) . . . no
N1 C8 H8' 111(2) . . . no
N1 C9 H9 109.2(13) . . . no
C10 C9 H9 111.3(15) . . . no
C15 C9 H9 110.6(15) . . . no
C12 C11 H11 122(2) . . . no
C10 C11 H11 120(2) . . . no
C11 C12 H12 116(3) . . . no
C13 C12 H12 125(3) . . . no
C14 C13 H13 120.4(19) . . . no
C12 C13 H13 120.7(19) . . . no
N2 C14 H14 116.5(19) . . . no
C13 C14 H14 121.4(19) . . . no
C15 C16 H16 122(3) . . . no
C17 C16 H16 119(3) . . . no
C18 C17 H17 117(3) . . . no
C16 C17 H17 123(3) . . . no
C17 C18 H18 126(3) . . . no
C19 C18 H18 115(3) . . . no
N3 C19 H19 116.2(17) . . . no
C18 C19 H19 121.6(17) . . . no
C21 C22 H22 116(3) . . . no
C23 C22 H22 124(3) . . . no
C22 C23 H23 116(4) . . . no
C24 C23 H23 123(4) . . . no
C23 C24 H24 120.2(3) 2_556 . . no
C23 C24 H24 120.2(3) . . . no

loop_
_geom_torsion_atom_site_label_1
_geom_torsion_atom_site_label_2
_geom_torsion_atom_site_label_3
_geom_torsion_atom_site_label_4
_geom_torsion
_geom_torsion_site_symmetry_1
_geom_torsion_site_symmetry_2
_geom_torsion_site_symmetry_3
_geom_torsion_site_symmetry_4
_geom_torsion_publ_flag
O2 Mn O1 C1 -87.5(2) . . . . no
O2 Mn O1 Mn 60.70(8) . . . 2_556 no
N1 Mn O1 C1 15.0(2) . . . . no
N1 Mn O1 Mn 163.20(8) . . . 2_556 no
N2 Mn O1 C1 -8.1(3) . . . . no
N2 Mn O1 Mn 140.13(14) . . . 2_556 no
```

```
N3 Mn O1 C1 86.5(2) . . . . no
N3 Mn O1 Mn -125.32(8) . . . 2_556 no
O1 Mn O1 C1 180.0(2) 2_556 . . . no
O1 Mn O1 Mn -31.84(7) 2_556 . . 2_556 no
O1 Mn O2 C20 -25.47(17) . . . . no
N1 Mn O2 C20 -111.09(16) . . . . no
N2 Mn O2 C20 179.34(18) . . . . no
N3 Mn O2 C20 168.16(15) . . . . no
O1 Mn O2 C20 52.62(17) 2_556 . . . no
O1 Mn N1 C7 32.62(14) . . . . no
O1 Mn N1 C8 -92.52(14) . . . . no
O1 Mn N1 C9 149.69(15) . . . . no
O2 Mn N1 C7 130.32(15) . . . . no
O2 Mn N1 C8 5.18(15) . . . . no
O2 Mn N1 C9 -112.61(14) . . . . no
N2 Mn N1 C7 -157.19(16) . . . . no
N2 Mn N1 C8 77.67(14) . . . . no
N2 Mn N1 C9 -40.12(14) . . . . no
N3 Mn N1 C7 -75.77(15) . . . . no
N3 Mn N1 C8 159.10(15) . . . . no
N3 Mn N1 C9 41.31(14) . . . . no
O1 Mn N1 C7 -6.1(3) 2_556 . . . no
O1 Mn N1 C8 -131.20(18) 2_556 . . . no
O1 Mn N1 C9 111.01(18) 2_556 . . . no
O1 Mn N2 C10 40.6(3) . . . . no
O1 Mn N2 C14 -122.9(2) . . . . no
O2 Mn N2 C10 124.70(17) . . . . no
O2 Mn N2 C14 -38.8(2) . . . . no
N1 Mn N2 C10 16.64(16) . . . . no
N1 Mn N2 C14 -146.9(2) . . . . no
N3 Mn N2 C10 -60.18(16) . . . . no
N3 Mn N2 C14 136.3(2) . . . . no
O1 Mn N2 C10 -149.21(15) 2_556 . . . no
O1 Mn N2 C14 47.3(2) 2_556 . . . no
O1 Mn N3 C15 -102.36(16) . . . . no
O1 Mn N3 C19 82.1(2) . . . . no
O2 Mn N3 C15 63.6(3) . . . . no
O2 Mn N3 C19 -111.9(2) . . . . no
N1 Mn N3 C15 -23.79(15) . . . . no
N1 Mn N3 C19 160.7(2) . . . . no
N2 Mn N3 C15 52.46(15) . . . . no
N2 Mn N3 C19 -123.1(2) . . . . no
O1 Mn N3 C15 178.71(15) 2_556 . . . no
O1 Mn N3 C19 3.2(2) 2_556 . . . no
O1 Mn O1 Mn 30.81(6) . . 2_556 2_556 no
O1 Mn O1 C1 -179.2(2) . . 2_556 2_556 no
O2 Mn O1 Mn -67.35(8) . . 2_556 2_556 no
O2 Mn O1 C1 82.66(19) . . 2_556 2_556 no
N1 Mn O1 Mn 70.31(18) . . 2_556 2_556 no
N1 Mn O1 C1 -139.67(19) . . 2_556 2_556 no
N2 Mn O1 Mn -145.02(7) . . 2_556 2_556 no
N2 Mn O1 C1 5.0(2) . . 2_556 2_556 no
N3 Mn O1 Mn 135.55(7) . . 2_556 2_556 no
N3 Mn O1 C1 -74.44(19) . . 2_556 2_556 no
Mn O1 C1 C2 148.59(18) . . . . no
Mn O1 C1 C6 -32.4(3) . . . . no
Mn O1 C1 C2 9.8(3) 2_556 . . . no
Mn O1 C1 C6 -171.22(16) 2_556 . . . no
Mn O2 C20 C21 172.58(10) . . . . no
Mn O2 C20 O2 -7.4(2) . . . 2_556 no
Mn N1 C7 C6 -70.0(2) . . . . no
C8 N1 C7 C6 56.1(3) . . . . no
C9 N1 C7 C6 179.3(2) . . . . no
Mn N1 C9 C10 61.1(2) . . . . no
Mn N1 C9 C15 -56.3(2) . . . . no
C7 N1 C9 C10 178.75(19) . . . . no
```

```
C7 N1 C9 C15 61.4(2) . . . . no
C8 N1 C9 C10 -58.0(2) . . . . no
C8 N1 C9 C15 -175.34(19) . . . . no
Mn N2 C10 C9 12.2(2) . . . . no
Mn N2 C10 C11 -167.52(19) . . . . no
C14 N2 C10 C9 177.6(2) . . . . no
C14 N2 C10 C11 -2.2(3) . . . . no
Mn N2 C14 C13 163.62(17) . . . . no
C10 N2 C14 C13 1.2(3) . . . . no
Mn N3 C15 C9 -.6(2) . . . . no
Mn N3 C15 C16 -176.42(18) . . . . no
C19 N3 C15 C9 175.5(2) . . . . no
C19 N3 C15 C16 -.3(3) . . . . no
Mn N3 C19 C18 175.36(17) . . . . no
C15 N3 C19 C18 .1(3) . . . . no
O1 C1 C2 C3 178.6(2) . . . . no
C6 C1 C2 C3 -.4(4) . . . . no
O1 C1 C6 C5 -179.7(2) . . . . no
O1 C1 C6 C7 -3.2(3) . . . . no
C2 C1 C6 C5 -.7(3) . . . . no
C2 C1 C6 C7 175.8(2) . . . . no
C1 C2 C3 C4 1.3(4) . . . . no
C2 C3 C4 C5 -1.0(4) . . . . no
C3 C4 C5 C6 -.1(4) . . . . no
C4 C5 C6 C1 .9(3) . . . . no
C4 C5 C6 C7 -175.5(2) . . . . no
C5 C6 C7 N1 -124.5(2) . . . . no
C1 C6 C7 N1 59.1(3) . . . . no
N1 C9 C10 C11 127.9(2) . . . . no
N1 C9 C10 N2 -51.9(3) . . . . no
N1 C9 C15 N3 40.8(3) . . . . no
C15 C9 C10 N2 66.9(3) . . . . no
C15 C9 C10 C11 -113.4(3) . . . . no
C10 C9 C15 C16 99.0(3) . . . . no
N1 C9 C15 C16 -143.4(2) . . . . no
C10 C9 C15 N3 -76.8(2) . . . . no
N2 C10 C11 C12 2.4(4) . . . . no
C9 C10 C11 C12 -177.3(2) . . . . no
C10 C11 C12 C13 -1.6(4) . . . . no
C11 C12 C13 C14 .7(4) . . . . no
C12 C13 C14 N2 -.5(4) . . . . no
C9 C15 C16 C17 -175.4(2) . . . . no
N3 C15 C16 C17 .1(3) . . . . no
C15 C16 C17 C18 .3(3) . . . . no
C16 C17 C18 C19 -.5(4) . . . . no
C17 C18 C19 N3 .3(4) . . . . no
O2 C20 C21 C22 31.18(17) . . . . no
O2 C20 C21 C22 -148.82(17) . . . 2_556 no
O2 C20 C21 C22 -148.82(17) 2_556 . . . no
C20 C21 C22 C23 179.6(2) . . . . no
C22 C21 C22 C23 -.4(3) 2_556 . . . no
C21 C22 C23 C24 .9(4) . . . . no
C22 C23 C24 C23 -.4(4) . . . 2_556 no

loop_
_geom_contact_atom_site_label_1
_geom_contact_atom_site_label_2
_geom_contact_distance
_geom_contact_site_symmetry_1
_geom_contact_site_symmetry_2
_geom_contact_publ_flag
Mn H2 2.86(3) . 2_556 no
O3 C16 3.418(7) . 4_555 no
O41 C18 3.340(15) . 1_565 no
O42 C22 3.282(13) . . no
O42 C23 3.173(14) . . no
```

O1 H7' 2.86(3) . . no
O2 H14 2.80(3) . . no
O2 H22 2.55(4) . . no
O3 H4 2.89(3) . 4_556 no
O3 H13 2.60(3) . . no
O3 H16 2.68(5) . 4_555 no
O41 H11 2.87(4) . 3_455 no
O41 H18 2.52(5) . 1_565 no
O42 H5 2.62(3) . 4_556 no
O42 H18 2.85(5) . 1_565 no
O42 H22 2.86(4) . . no
O42 H23 2.55(5) . . no
N2 H2 2.91(3) . 2_556 no
N2 H8 2.78(3) . . no
N3 H7' 2.58(3) . . no
C1 C8 3.528(3) . . no
C4 C17 3.563(4) . 4_556 no
C4 C13 3.469(4) . 4_546 no
C4 C12 3.586(4) . 4_546 no
C5 C17 3.512(4) . 4_556 no
C5 C12 3.573(4) . 4_546 no
C5 C13 3.308(4) . 4_546 no
C8 C17 3.531(3) . 4_556 no
C8 C1 3.528(3) . . no
C12 C5 3.573(4) . 4_556 no
C12 C17 3.405(3) . 4_555 no
C12 C4 3.586(4) . 4_556 no
C13 C17 3.439(3) . 4_555 no
C13 C5 3.308(4) . 4_556 no
C13 C4 3.469(4) . 4_556 no
C16 O3 3.418(7) . 4_545 no
C17 C5 3.512(4) . 4_546 no
C17 C12 3.405(3) . 4_545 no
C17 C8 3.531(3) . 4_546 no
C17 C13 3.439(3) . 4_545 no
C17 C4 3.563(4) . 4_546 no
C18 O41 3.340(15) . 1_545 no
C22 O42 3.282(13) . . no
C23 O42 3.173(14) . . no
C1 H8" 2.86(3) . . no
C3 H17 3.06(5) . 4_556 no
C4 H13 2.97(4) . 4_546 no
C4 H17 2.70(5) . 4_556 no
C5 H17 2.88(5) . 4_556 no
C5 H13 3.02(4) . 4_546 no
C5 H8" 3.01(4) . . no
C6 H8" 2.39(4) . . no
C10 H8 2.54(3) . . no
C11 H4 3.03(4) . 1_554 no
C12 H17 3.01(4) . 4_555 no
C13 H17 2.95(4) . 4_555 no
C13 H5 3.08(4) . 4_556 no
C14 H2 3.07(3) . 2_556 no
C14 H3 2.98(4) . 1_554 no
C15 H7' 2.47(3) . . no
C16 H8" 2.97(4) . 4_546 no
C17 H12 3.07(4) . 4_545 no
C17 H8" 2.70(3) . 4_546 no
C18 H12 3.09(4) . 4_545 no
C22 H7 2.85(3) . 4_556 no
C22 H9 3.07(3) . 3_455 no
C23 H8' 2.94(3) . 4_556 no
C23 H9 2.92(3) . 3_455 no
C23 H7 3.05(3) . 4_556 no
C24 H8' 2.77(3) . 3_455 no
C24 H8' 2.77(3) . 4_556 no

```
H2 Mn 2.86(3) . 2_556 no
H2 H3 2.51(5) . 2_557 no
H2 N2 2.91(3) . 2_556 no
H2 C14 3.07(3) . 2_556 no
H3 H2 2.51(5) . 2_557 no
H3 C14 2.98(4) . 1_556 no
H4 O3 2.89(3) . 4_546 no
H4 C11 3.03(4) . 1_556 no
H5 C13 3.08(4) . 4_546 no
H5 O42 2.62(3) . 4_546 no
H5 H7 2.27(4) . . no
H7 H8' 2.55(5) . . no
H7 H9 2.47(3) . . no
H7 H5 2.27(4) . . no
H7 C23 3.05(3) . 4_546 no
H7 C22 2.85(3) . 4_546 no
H7' N3 2.58(3) . . no
H7' O1 2.86(3) . . no
H7' C15 2.47(3) . . no
H8 N2 2.78(3) . . no
H8 C10 2.54(3) . . no
H8' C23 2.94(3) . 4_546 no
H8' C24 2.77(3) . 4_546 no
H8' H7 2.55(5) . . no
H8' H9 2.48(4) . . no
H8' C24 2.77(3) . 3_545 no
H8" C17 2.70(3) . 4_556 no
H8" C5 3.01(4) . . no
H8" C6 2.39(4) . . no
H8" C1 2.86(3) . . no
H8" C16 2.97(4) . 4_556 no
H9 H7 2.47(3) . . no
H9 H8' 2.48(4) . . no
H9 H11 2.46(4) . . no
H9 H16 2.49(5) . . no
H9 C22 3.07(3) . 3_545 no
H9 C23 2.92(3) . 3_545 no
H11 H9 2.46(4) . . no
H11 O41 2.87(4) . 3_545 no
H12 C17 3.07(4) . 4_555 no
H12 C18 3.09(4) . 4_555 no
H13 O3 2.60(3) . . no
H13 C5 3.02(4) . 4_556 no
H13 C4 2.97(4) . 4_556 no
H14 O2 2.80(3) . . no
H16 O3 2.68(5) . 4_545 no
H16 H9 2.49(5) . . no
H17 C4 2.70(5) . 4_546 no
H17 C3 3.06(5) . 4_546 no
H17 C12 3.01(4) . 4_545 no
H17 C13 2.95(4) . 4_545 no
H17 C5 2.88(5) . 4_546 no
H18 O41 2.52(5) . 1_545 no
H18 O42 2.85(5) . 1_545 no
H22 O2 2.55(4) . . no
H22 O42 2.86(4) . . no
H23 O42 2.55(5) . . no

loop_
_geom_hbond_atom_site_label_D
_geom_hbond_atom_site_label_H
_geom_hbond_atom_site_label_A
_geom_hbond_distance_DH
_geom_hbond_distance_HA
_geom_hbond_distance_DA
_geom_hbond_angle_DHA
```

```
_geom_hbond_site_symmetry_A
_geom_hbond_publ_flag
#
#D H A D - H H...A D...A D - H...A symm(A)
#
C7 H7' N3 .98(3) 2.58(3) 3.183(3) 120(2) . yes
C13 H13 O3 .94(4) 2.60(3) 3.465(8) 154(3) . yes
C22 H22 O2 .81(3) 2.55(4) 2.839(4) 103(3) . yes
C23 H23 O42 .81(5) 2.55(5) 3.173(14) 135(5) . yes

# End of Crystallographic Information File
```